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| <b>(54) Title:</b> ALCOHOLIC, READY-TO-FREEZE BEVERAGE<br><br><b>(57) Abstract</b><br><br>A ready-to-freeze, packaged alcoholic beverage having a pH of about 3.0 - about 5.0 includes an alcohol, sugar, a flavoring, water, a stabilizer blend of a locust bean gum, guar gum and, optionally, pectin, and other ingredients. The beverage is shelf-stable and can be frozen in a freezer to produce a frozen or semi-frozen, slushy cocktail which retains its consistency for a prolonged time period. Also disclosed is a container for packaging the beverage.   |           |   |

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ALCOHOLIC, READY-TO-FREEZE BEVERAGE  
CROSS-REFERENCE TO RELATED APPLICATIONS

This is a continuation-in-part of U.S. Patent Application, Serial Number 08/321,885, filed  
5 October 14, 1995.

BACKGROUND OF THE INVENTION

(a) FIELD OF THE INVENTION

This invention relates to a ready-to-freeze alcoholic beverage (or "alcoholic, ready-to-freeze  
10 beverage"). More particularly, the invention is directed to such an alcoholic beverage which is shelf stable in liquid form and designed to be placed in a freezer, preferably a consumer's freezer, for a period of time until it assumes a "slushy"  
15 consistency like a frozen cocktail prepared in a blender or commercial slush machines found in bars and restaurants.

The invention is also directed to a container for packaging the beverage.

20 (b) DESCRIPTION OF RELATED ART

Frozen cocktails continue to be popular bar and home beverages. Products have been introduced to ease the preparation of frozen drinks in the home. However, for the most part, consumers must add a  
25 specific alcohol type and deal with the inconvenience of having complex preparation knowledge, proper equipment, and clean up.

Prior art sought to provide both alcoholic and non-alcoholic beverages capable of forming a  
30 crystalline structure upon being placed in the consumer's freezer. For example, Ashmont et al., U.S. Patent No. 4,790,999, discloses a ready to consume alcoholic beverage which forms a soft ice at freezer temperatures. A critical component of this  
35 beverage is carboxymethyl cellulose ("CMC"), which is

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known for its compatibility in pH systems of 4.0-10.0. Ashmont's beverage is believed to have pH of about 3.0 or lower. Ashmont teaches the importance of using CMC at an amount ranging from 0.020% by weight to about 0.1% by weight.

Marulich, U.S. Patent No. 3,826,829, discloses a substantially non-alcoholic mixture for a shelf stable carbonated or non-carbonated beverage which can be consumed after conventional chilling or placed in a freezer and consumed as a soft frozen carbonated beverage. The carbonated beverage is made by incorporating a pectin stabilizing system and a gaseous phase into an aqueous composition including water, sugars, polyols, flavor and color agents to form a stable carbonated beverage.

LeVan, U.S. Patent No. 3,619,205, discloses a method for preparing beverage products that can be served as a drinkable slush ice composition. The slush is made by mixing a frozen, ground, homogeneous blend of (a) about one part of a mixture of sucrose syrup, flavor, an edible food acid, a water dispersible gum, an edible polyhydric alcohol, and water, with (b) about 0.25 parts of flaked ice, having particle sizes ranging from about 250 to 2,000 microns, and refrigerating the mixture at -10°F. LeVan's product is believed to have pH of 2.5. Among the various water-dispersible gums and colloids which may be utilized are gum tragacanth, gum karaya, gum arabic, locust bean gum, guar seed gum, carrageenan, pectin, CMC, alginates, gelatin, gum ghatti, agar, and the like. Example 1 discloses CMC in a range of 0.5 to 1.0% by weight.

Homler et al., U.S. Patent No. 3,897,571, discloses a spoonable slush concentrate containing an ionic cellulose gum, such as CMC or its equivalent,

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to prevent separation at elevated storage temperatures as high as 15-20°F. The concentrate is spoonable at freezer temperatures and can be mixed with either water, milk or alcohol to form a slush.

5 In obtaining the slush it is important that the gums used in the invention are fully hydrated and dispersed, and therefore, in the method of preparing the spoonable concentrate of the invention, the gums are preferably hydrated and dispersed prior to  
10 addition of the sugar solids. After the gums are hydrated and mixed with the sugar solids, flavors and acids are added when used. The concentrate is then subjected to a commercial sterilization or pasteurization at a temperature of about 160°F. The  
15 concentrate is then cooled to about 27-32°F preparatory to crystallization. Freezing point depressants such as acids and alcohols may then be added. This reduces the concentrate's ice point and therefore makes it more spoonable at 0°F. The  
20 concentrate is then crystallized. After crystal development, the crystal slush is transferred under control temperatures not to exceed 20°F to a holding vessel wherefrom the slush concentrate is discharged into a container and packaged according to technology  
25 known in the art. The filled container is then blast frozen at -10°F or below to a "center" temperature of 0°F or below, the center being the geometric center of the mass.

Van den Hoven et al., U.S. Patent No. 5,066,509,  
30 discloses a liqueur or alcohol-containing beverage comprising cream, yogurt or another dairy product, obtained by mixing the dairy product with alcohol or an alcohol-containing liquid, flavoring agents, coloring agents, emulsifiers, stabilizers, sugars or  
35 artificial sweetening agents, fats, acids and further

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conventional ingredients (col. 1, lines 8-14). The stable dairy liquor can be stored in cool and uncool conditions for considerable time if the fats used in the preparation are mainly saturated fatty acids having 6 to 12 carbon atoms (col. 3, lines 34-39). Suitable stabilizers are pectin and CMC.

Daher et al., U.S. Patent No. 4,738,857, discloses a container for packaging an alcoholic beverage containing essential oils. The container includes an interior layer, in contact with the alcoholic beverage, and a second layer. The interior layer consists essentially of polypropylene homopolymer. The second layer, located exterior to the outside layer, is composed of an oxygen barrier polymer, such as ethylene vinyl alcohol copolymer. The container is particularly useful in connection with the alcoholic soft ice composition of Ashmont et al., U.S. Patent No. 4,790,999.

While the art discussed above provides important advantages, none of it provides a ready-to-freeze alcoholic beverage having the combination of higher pH, excellent shelf stability, taste and, once frozen, the ability to maintain its semi-frozen, slushy consistency or state for a relatively prolonged time period.

The art also has not provided a container suitable for packaging beverages, such as the ready-to-freeze alcoholic beverage of the invention, which has a strong seal strength and is substantially resistant to breakage if it is dropped in transit.

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SUMMARY OF THE INVENTION

The invention provides a ready-to-freeze, alcoholic beverage comprising water, at least one sugar, a flavoring, a beverage alcohol, and a stabilizing system which includes guar gum and locust bean gum, and optionally, pectin.

The alcoholic beverage may also include common acidulants, such as citric acid. The alcoholic beverage has a pH of about 3.0 to about 5.0.

The sugar comprises sucrose, fructose and dextrose. Alternatively, the sugar may comprise sucrose and high fructose corn syrup ("HFCS") which includes dextrose, fructose, maltose and higher saccharides. The ready-to-freeze alcoholic beverage has a proof of about 6° to about 28° (which is equivalent to the ethyl alcohol content of about 3.0 to about 14% wt.). The stabilizing system, also referred to herein as the "stabilizer blend", is present in the beverage in an amount ranging from about 0.04% to about 0.13% by weight.

The beverage may also contain other ingredients, such as one or more clouding agent, a preservative and more than one flavoring.

The ready-to-freeze alcoholic beverage forms a slushy, fine crystalline structure at freezer temperatures (about -5 to about 20°F) after being placed in the freezer for a period of about 3 to about 6 hours.

The invention is also directed to a container, preferably in the form of a flexible plastic pouch, comprising one of the following multi-layered structures:

Structure A

- (a) polyester film;
- (b) aluminum foil;

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- (c) polyester film;
- (d) linear low density polyethylene film.

Structure B

- 5 (a) polyvinylidene chloride coated  
biaxially oriented Nylon or any other  
Nylon film;
- (b) Nylon/ethylene/vinyl alcohol co-  
extruded layer film;
- (c) linear low density polyethylene film.

10

Structure C

- (a) Nylon film;
- (b) aluminum foil;
- (c) copolymer of ethylene and propylene.

15 Suitable materials which would adhere respective  
film layers may be used between layers of the  
container. Such materials are exemplified by  
adhesives, e.g., polyester adhesives.

20 As pointed out in greater detail below, the  
alcoholic beverage of this invention provides  
important advantages. With the beverage of this  
invention the consumer is not inconvenienced by the  
necessity of knowing the recipe, having the required  
ingredients on hand, having the proper equipment,  
such as a blender, or cleaning the equipment. The  
25 beverage of the invention may be packaged in a single  
serve container which also allows consumers to  
"switch" flavors without having to make up a large  
batch of a beverage in a blender and potentially  
throw away any unconsumed portion. The alcoholic  
30 beverage of the invention is formulated at a  
considerably higher pH and utilizes a specific and  
complex blend of stabilizing ingredients to give it a  
superior and much preferred crystalline ice structure  
and, thus taste, as compared to prior art products.



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The container of the invention also provides important advantages. The container exhibits an excellent seal strength and aids the beverage of the invention in achieving a long shelf life.

5 DETAILED DESCRIPTION OF THE INVENTION

The term "ready-to-freeze" alcoholic beverage means that the beverage can be frozen, e.g., in a consumer's freezer, as purchased substantially without any other preparation, modification, blending  
10 or additions of other ingredients thereto to produce the frozen or semi-frozen slushy cocktail. For example, it is not necessary to add ice.

It is important that the alcoholic beverage has pH of about 3.0 to about 5.0, preferably about 3.2 to  
15 about 4.0, more preferably about 3.3 to about 3.8 and most preferably about 3.4 to about 3.7. At these ranges, optimum taste can be achieved based on sweetener levels of the beverage formula - i.e. more acid used with sweeter beverages, less acid used with  
20 lower sugar content beverages. Those pH ranges are achieved by the addition of commonly used acidulants such as citric acid, malic acid, tartaric acid and fumaric acid. The reason(s) for the importance of these pH ranges is not understood. However, without  
25 wishing to be bound by any operability theory, it is believed that at pH's less than 3.0 the hydrative rate of the stabilizers used in the beverage diminishes, thereby affecting the stabilizers' ability to control the ice crystal structure of the  
30 water phase.

The water used in the beverage is deionized water.

The sugar balance of the beverage has been found in taste panels to be important in arriving at  
35 optimum taste and texture. To accomplish this, the

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beverage comprises at least one or more of the following sweeteners: sucrose, fructose, or dextrose. Alternatively, the beverage may preferably comprise a mixture of sucrose and high fructose corn syrup, which includes dextrose, fructose, maltose and higher saccharides. The final beverage product has a total sugar content as measured in BRIX (or "Brix") of about 12° to about 19°. The amounts of the individual sugars used are: sucrose, about 7.4 to about 8.5, preferably about 7.6 to about 8.3, and most preferably about 7.8 to about 8.1% by weight (wt.); fructose, about 3.1 to about 4.3, preferably about 3.3 to about 4.0, and most preferably about 3.5 to about 3.8% by weight; dextrose, about 3.9 to about 5.0, preferably about 4.1 to about 4.8, and most preferably about 4.3 to about 4.6% by weight. If the mixture of sucrose and high fructose corn syrup is used, the amounts of the individual sugar components are: sucrose, about 7.4 to about 8.5, preferably about 7.6 to about 8.3, and most preferably about 7.8 to about 8.1% by weight; and high fructose corn syrup, about 8.1 to about 9.3, preferably about 8.3 to about 9.0, and most preferably about 8.5 to about 8.8% by weight. The high fructose corn syrup includes about 48 to about 52% by weight dextrose, about 40 to about 44% by weight fructose, about 0.5 to about 2.0% by weight maltose, and about 3 to about 7% by weight higher saccharides.

Any alcohol beverage suitable for human consumption can be utilized as the source of the beverage alcohol, e.g., grain neutral spirits, vodka, whisky, rum, tequila, malt beverage, wine, such as Other Than Standard ("OTS") orange wine, and similar beverages. The preferred alcohol beverage used in the ready-to-freeze alcoholic beverage is OTS orange

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wine. The term "Other Than Standard" wine ("OTSW"), as is known in the industry, designates a beverage comprising a minimum of 5% standard wine combined with fruit spirits and water, to deliver about 20 -  
5 about 22% wt. ethyl alcohol and a minimum 20 ppm of fusel oil. The term "beverage alcohol", as is known to those skilled in the art, means an alcohol suitable for human consumption. Such an alcohol usually includes primarily ethyl alcohol (about  
10 99.99% wt.), and minor amounts of other alcohols, such as propyl alcohol, isopropyl alcohol and other alcohols. As is known to those skilled in the art, flavorings may also contain ethyl alcohol. The ethyl alcohol content of flavorings should be considered in  
15 producing the ready-to-freeze alcoholic beverage of a particular ethyl alcohol content.

The ready-to-freeze alcoholic beverage has a proof of about 6° to about 28°, preferably about 7 to about 28° (equivalent to the ethyl alcohol content of  
20 about 3.5 to about 14%), more preferably about 8 to about 14° (equivalent to the ethyl alcohol content of about 4 to about 7% wt.), and most preferably about 11 to about 13° (equivalent to the ethyl alcohol content of about 5.5 to about 6.5% wt.). The  
25 alcoholic content of the ready-to-freeze alcoholic beverage has a significant impact on the freezing properties of the beverage. Lower amounts of beverage alcohol decrease the beverage's resistance to freezing, thereby producing a product which may  
30 freeze prematurely and more solidly, which is undesirable. Increasing the alcohol level may result in resistance to freezing and may require lower temperatures to freeze.



The content of the remaining ingredients of the  
35 alcoholic beverage composition of the invention can

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be adjusted so that the beverage, when subjected to the freezer conditions for the time periods set forth herein, produces a slushy, frozen cocktail product. The term "slushy frozen cocktail product" means that

5 the product has the consistency of a partly melted, semi-frozen soft ice, which is neither freely pourable nor hard like an ice cube. The product is relatively easily deformable, e.g., by application of finger pressure, and can be easily manipulated or

10 removed from the container with a hand-operated utensil, such as a spoon. Some well known examples of such slushy frozen cocktails are frozen margarita and pina colada. This invention provides a beverage product which would freeze to produce the slushy

15 frozen cocktail in substantially every consumer's freezer, specifically in a wider temperature range than afforded by prior art, i.e., about -5°F to about 20°F.

The relatively low proof of the beverage of the invention makes the choice of the stabilizing system especially critical to deliver the desired type frozen drink consistency imitating that produced in a blender.

In one embodiment, a stabilizing system

25 comprised of the combination (or blend) of guar gum and locust bean gum produces the alcoholic beverage of the invention having excellent properties. This combination of ingredients produces a beverage product which freezes well, has an excellent texture,

30 small ice crystals, substantially no undesirable ice crystal sheets, and delivers unexpected benefits in enabling the alcoholic beverage product to remain in a frozen or semi-frozen state at room temperature, once removed from the freezer, for a period of about

35 20-30 minutes. To further enhance the texture, in

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another embodiment a small amount of pectin is preferably added to the guar gum and locust bean gum stabilizer blend. The pectin, in proper proportions, has an unexpected beneficial impact on further  
5 enhancing the amount of crunch (while maintaining a substantial absence of ice crystal sheets) and delivering a beverage product substantially identical to a blender frozen drink.

In one embodiment, the alcoholic, ready-to-  
10 freeze beverage composition includes the following ingredients as the stabilizer blend: guar gum, about 0.005 to about 0.5, preferably about 0.010 to about 0.30, and most preferably about 0.016 to about 0.020% by weight and locust bean gum, about 0.025 to about  
15 0.090, preferably about 0.030 to about 0.060, and most preferably about 0.045 to about 0.055% by weight.

In another embodiment, the alcoholic beverage composition includes the following ingredients as the  
20 stabilizer blend: locust bean gum, about 0.025 to about 0.090, preferably about 0.030 to about 0.060, and most preferably about 0.045 to about 0.055% by weight, guar gum about 0.005 to about 0.50, preferably about 0.010 to about 0.3, and most  
25 preferably about 0.016 to about 0.020% by weight, and pectin about 0.003 to about 0.1, preferably about 0.003 to about 0.075, more preferably about 0.003 to about 0.05, most preferably about 0.003 to about 0.03% by weight of the beverage. The pectin is  
30 preferably low methoxy pectin ("LMP"). In one specifically preferred embodiment, the alcoholic, ready-to-freeze beverage comprises a stabilizer blend which includes about 0.05% by weight of locust bean gum and about 0.018% by weight of guar gum. In  
35 another specifically preferred embodiment, the

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alcoholic, ready-to-freeze beverage composition comprises a stabilizer blend which includes about 0.005% by weight of pectin, about 0.05% by weight of locust bean gum and about 0.018% by weight of guar gum. In both of these embodiments, the alcoholic beverage of the invention has pH of about 3.5 to about 3.7 and has proof of about 10 to about 14.

All amounts of all ingredients set forth herein are calculated on the basis of the entire beverage formulation.

In one embodiment, the stabilizer blend consists essentially of locust bean gum and guar gum in the relative amounts set forth above. In another embodiment, the stabilizer blend consists essentially of pectin, such as LMP, locust bean gum and guar gum in the relative amounts set forth above.

This unusual stabilizer blend is believed to have excellent properties in controlling ice crystal growth in a frozen product which ultimately improves the finished product texture and mouth feel. Without wishing to be bound by any theory of operability, it is believed that the crystalline structure is maintained in the frozen state by allowing re-absorption of the water/alcohol molecules and re-dispersion of ingredients during the thaw cycles common to consumer freezers.

It is also believed that the blend of the stabilizing system serves to protect the ice crystal structure for extended periods in a freezer. The beverage, once frozen, maintains its frozen or partially frozen, slushy state for a surprisingly extended time, e.g., 20-30 minutes at room temperature.

The amount of individual ingredients of the stabilizer blend and of relative proportions of the

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ingredients to each other may be varied within the ranges set forth herein to obtain the alcoholic, ready-to-freeze beverage having desired properties and characteristics, such as taste characteristics.

- 5 The amounts and/or the proportions of various ingredients of the stabilizer blend may also be adjusted within the disclosed ranges in view of availability of various ingredients and/or to produce the most cost-effective alcoholic, ready-to-freeze  
10 beverage.

Based on results of taste panels' evaluations, the beverage of this invention, when frozen, delivers a substantially improved crystalline structure (small  
15 crystal size, substantial absence of ice crystal sheets, pleasant mouthfeel) and greatly improved reduced rate of melt, as compared to prior art formulations.

The unusual blend of stabilizers at the pH levels of the beverage requires the use of an equally  
20 unusual process to produce the beverage. While prior art required heating to achieve commercial sterility, this invention requires a heating phase which is only necessary to achieve the formulation of the alcoholic beverage of the invention. Therefore commercial  
25 sterility temperatures are not required or needed.

It is believed that the heating phase activates the stabilizer blend which is ultimately responsible for maintaining and delivering the fine crystalline, frozen blender-like texture. To carry out the  
30 heating phase (or a "cook phase"), the stabilizer blend is first blended with a dry dispersant in a ratio of approximately 3 parts dispersant to 1 part by weight stabilizer blend and then added, preferably via high shear agitation, to a portion of th  
35 deionized water phase. Suitable dispersants are the

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dry sugar or sugars used in the beverage, such as sucrose, fructose or dextrose. Only a portion of the dry sugar is used in this blending step. The portion of the dry sugar used is about 5- about 20%,  
5 preferably about 10 - about 15% of the total dry sugar used for the composition. About 15 - about 30%, preferably about 20 - about 25% of the water phase is used in the blending phase. The remainder of the dry sugar is then added to produce a  
10 sugar/stabilizer slurry. This slurry is heated to a temperature in the range of about 149° to about 190°F, combined with a portion of the deionized water to cool the slurry to about 100°F and then added to the balance of the beverage formulation.

15 While similar heating steps may have been used in the dairy industry, the cook phase of this invention is not believed to have been suggested for the alcoholic beverages of this type in the past.

The water phase is defined as the deionized  
20 water required to produce the formulation.

Common acidulants, such as citric acid, malic acid, tartaric acid, fumaric acid and preferably citric acid, are used in the beverage formulation to achieve the optimal taste expectations. The  
25 acidulants are used in the amounts of about 0.05 to about 0.7, preferably about 0.2 to about 0.4% by weight. The approximate range of citric acid, one of the preferred acidulants, used in this invention is about 0.10 to about 0.70% by weight.

30 Also used to deliver taste requirements is at least one flavoring included in the beverage of the invention in the range of about 1.0 to about 12.0, preferably about 3.0 to about 9.0, and most preferably about 4.0 to about 7.0% by weight.



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A flavoring may be any suitable material which will impart a desired flavor to the beverage of the invention. Examples of suitable flavorings include commercial flavors, naturally derived flavoring materials, such as herbs, spices, and artificial  
5 flavoring materials, such as vanilin, phenylethyl acetate, and ethyl acetate.

In one preferred embodiment for making a flavoring, the flavoring is made by adding one or  
10 more commercial flavors to a source of a beverage alcohol, such as grain neutral spirits, citric acid and water. The amount of the source of a beverage alcohol is such that the final alcoholic, ready-to-freeze beverage, including additional amounts of  
15 beverage alcohol, if needed, has the proof set forth in this disclosure. The amount of the citric acid is such that the beverage of the invention, which may include additional amounts of citric acid or other acidulants as discussed herein, has pH levels also  
20 defined in the disclosure. Suitable commercial flavors include one or more essential oils in small amounts, alone or in combination with natural or artificial flavors, or juice concentrates. The types of flavorings used and the amounts thereof are those  
25 which are necessary to achieve the desired flavor of the beverage, e.g., pina colada, frozen margarita.

Other ingredients commonly used in the food industry in the beverages of this type may also be utilized in the beverage. Such ingredients include:  
30 clouding agents (which are typically comprised of the following ingredients: modified food starch, medium chain triglycerides, glycerol, partially hydrogenated soybean oil, brominated vegetable oil, citric acid, potassium sorbate, sodium benzoate and natural  
35 tocopherol), preservatives, such as sodium benzoate

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and potassium sorbate, and colors or colorants. The relative amounts of such other ingredients will be such as are necessary to perform their respective functions. For example, the clouding agents are used  
5 in the amount of about 0.01 to about 0.20% wt. The preservatives are used in the amount of about 0.03 to about 0.06% wt.

However, clouding aspects are not an essential element of producing the invention. The clouding  
10 agents may be omitted from the composition of the alcoholic, ready-to-freeze beverage without adversely affecting the taste or substantially any other preferred properties of the beverage. If clouding agents are deleted, a substantially clear alcoholic,  
15 ready-to-freeze beverage will be obtained, instead of a cloudy beverage. Therefore, clouding agents affect only the visual appearance of the beverage of the invention.

Similarly, preservatives, such as sodium  
20 benzoate and potassium sorbate, are not necessary to produce the beverage of the invention, having the preferred properties described herein. For example, if preservatives are not permitted to be used in beverages of this type due to regulatory  
25 restrictions, they can be deleted without sacrificing quality of the product or any of its preferred properties. As would be readily appreciated by those skilled in the art, the preservatives are incorporated into the beverage of the invention  
30 primarily for microbiological control, i.e., to retard or prevent microbiological growth which may lead to spoilage during distribution of the beverage. If preservatives are not used, the microbiological  
c ntr l may be provided by pasteurization, packaging  
35 under sterile conditions or some other means of

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retarding or preventing microbiological growth in the beverage.

The formulated alcoholic beverage can be packaged for sale in any suitable container, i.e.,  
5 glass, rigid plastic, flexible plastic pouch, metal, laminated paperboard or a combination of any of these. One preferred method is to package the product in a laminated flexible plastic pouch of 8.125 inch x 4.75 inch in size in which 200 ml to 240  
10 ml of the alcohol beverage product will be contained. The pouch is filled using a form-fill-seal machine; for example, a Prodo Pak or Bartelt, and, if desired, 36 to 45 pouches are packed per shipcase. The pouch may have the construction of one of the structures A,  
15 B or C summarized below. However, it is preferred that the pouch have the construction of the structures A or B. The first layer or film of each container described below is the outer film and is reverse gravure printed, and the last layer or film  
20 is in contact with the alcoholic beverage. The dimensions in the structures summarized below refer to thickness of the layers.

Structure A

- 25 Layer 1. About 0.00040 inch to about 0.00060 inch polyester;  
Layer 2. About 0.0004 inch to about 0.0006 inch aluminum foil;  
Layer 3. About 0.00040 inch to about 0.00060 inch polyester;  
30 Layer 4. About 0.0020 inch to about 0.0040 inch linear low density polyethylene or linear low density polyethylene which includes octene and ethylene.

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Structure B

Layer 1. About 0.0060 inch to about 0.0080 inch polyvinylidene chloride coated biaxially oriented Nylon;

5 Layer 2. About 0.0009 inch to about 0.0011 inch Nylon/ethylenevinyl alcohol coextruded;

Layer 3. About 0.0020 inch to about 0.0030 inch linear low density polyethylene (homopolymer).

10 In Structure B, layer 1 may be substituted by any other Nylon, and layer 2 by any Nylon, coated or uncoated. Layer 3 in Structure B may be any linear low density polyethylene homopolymer.

Structure C

15 Layer 1. About 0.0005 to about 0.0007 inch biaxially oriented Nylon;

Layer 2. About 0.0006 to about 0.0008 inch aluminum foil;

20 Layer 3. About 0.0020 to about 0.0040 inch copolymer of ethylene and propylene and/or polyethylene.

If necessary, in Structure C, suitable adhesives are used between layers 1 and 2 and 2 and 3. For example, 2.5 lbs Morprime adhesive may be used between layers 2 and 3. In one preferred embodiment, 25 the multi-layer container of the above construction holds about 200 - about 240 ml of the beverage. Different materials may be used for the various layers of the container of Structure C. For example, the third layer may be made of linear low density 30 polyethylene, and the second layer of a metalized Nylon or polyester film. In all embodiments of the invention directed to the container, any material which would provide adhesion between plastic films of the container to provide secure lamination between 35 the films, so that the resulting container would

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withstand shipping, can be used. Suitable materials are any of the well known polyester adhesives or polyolefins.

5 The plastic pouches when packaged in multi-packs provide multiple servings of the frozen or semi-frozen slushy cocktail. The plastic pouches of any of the structures described above are made by conventional methods known to those skilled in the art, for example, by any form-fill-seal machine.

10 While the container of the invention is preferably used to package the beverage of the invention, it can be used for packaging a variety of any goods, including food products or any beverages.

15 After the frozen beverage is removed from the freezer, the consumer massages the package gently and pours it into a suitable container, such as a glass for a frozen type cocktail. It can also be spooned out of its container. Once frozen, the slushy cocktail can be removed from the freezer at any time  
20 and consumed.

The following Examples further illustrate the essential features of the invention. However, it will be apparent to those skilled in the art that the specific reactants and reaction conditions used in  
25 the examples do not limit the scope of the invention. In the examples, a reference to % by weight (or % wt.) of alcoholic beverage or % wt. alcohol indicates % wt. of ethyl alcohol in the beverage.

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EXAMPLES 1A-1B  
(Beverage of the Invention)

|    |   | <u>Example</u> |                                      |  |
|----|---|----------------|--------------------------------------|--|
|    | <u>INGREDIENTS (% BY WEIGHT)</u>        | <u>1A</u>      | <u>% by weight</u><br><u>alcohol</u> | <u>1B</u> <u>% by weight</u><br><u>alcohol</u> |
| 5  | OTHER THAN STANDARD                     |                |                                      |  |
|    | ORANGE WINE (821% ETHYL ALC.)           | 19.71          | 4.139                                | 19.92 4.183                                    |
|    | SUCROSE                                 | 7.90           |                                      | 7.99   |
|    | HIGH FRUCTOSE CORN SYRUP                |                |                                      |  |
|    | DEXTROSE (50%)                          | 4.39           |                                      | 4.44   |
|    | FRUCTOSE (42%)                          | 3.68           |                                      | 3.73   |
|    | MALTOSE (1.5%)                          | 0.132          |                                      | 0.133  |
|    | HIGHER SACCHARIDES (5.0%)               | 0.438          |                                      | 0.444  |
|    | CITRIC ACID                             | 0.253          |                                      | 0.350  |
|    | SODIUM CITRATE                          | 0.16           |                                      | 0.165  |
| 10 | STABILIZER BLEND                        | 0.089          |                                      | 0.090  |
|    | FLAVORING                               | 5.49           | 1.293                                | 5.524 1.636                                    |
|    | DRY COLOR                               | 0.00036        |                                      | 0.0144   |
|    | CLOUDING AGENT                          | 0.018          |                                      | 0.157  |
|    | PRESERVATIVES                           | 0.047          |                                      | 0.047  |
|    | (POTASSIUM SORBATE AND SODIUM BENZOATE) |                |                                      |  |
|    | BALANCE, DEIONIZED WATER TO 100%        |                |                                      |  |
| 15 | TOTAL % ALCOHOL BY WEIGHT               |                | 5.432                                | 5.819  |

Example 1A yields a 5.432% wt. alcoholic beverage with a pH of 3.5. Example 1B yields a 5.819% wt. alcoholic beverage with a pH of 3.4.

20 The beverage of Example 1A had the flavor of pineapple; the beverage of Example 1B had the flavor of a frozen margarita.

The stabilizer blend in Examples 1A and 1B was comprised of 0.05% by weight of locust bean gum, 25 0.018% by weight of guar gum and 0.005% by weight of pectin and a filler (such as sugar, salt or whey) as balance. Additional ingredients included: a clouding agent and preservatives, such as sodium benzoate and potassium sorbate. The flavoring was 30 made as discussed above for the preferred embodiment, i.e., by adding one or more commercial flavors to grain neutral spirits, citric acid and water. The content of flavoring specified above therefore refers to the amount of the resulting mixture of the 35 flavors, grain neutral sprits, citric acid and water.

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To prepare the above formulation, the solutions of citric acid, sodium citrate and preservatives were first blended and set aside. Next, the stabilizer blend was dispersed with 15% of the sucrose in a 3:1 ratio of the sucrose to stabilizer blend and added via high shear mixing to 25% of deionized water. The remainder of the sucrose was then added to the product of the blending step to form a sugar/stabilizer slurry. This slurry was then heated to approximately 165°F to activate the stabilizer blend. Once heated, the sugar stabilizer slurry blend was transferred to a large blend tank, cooled to a temperature of about 100 °F, and then the balance of the formula ingredients was added to the tank in the following order: balance of deionized water, preservatives, high fructose corn syrup, clouding agent or cloudifier, other than standard orange wine, citric acid, sodium citrate, flavors. Once packaged in a flexible pouch container, the above formula froze in a home freezer in about 3-4 hours; the pouch was removed from the freezer and massaged, the product appearance and taste was that of a refreshing frozen cocktail with a very fine crystalline structure.

25

EXAMPLES 2A-2B  
(Beverage of the Invention)

A frozen flavored cocktail was prepared following substantially the same process and using substantially the same ingredients as in Example 1A and 1B, but utilizing an 80° beverage alcohol. Thus, the stabilizer blend, the flavors and the clouding agent were the same and used in the same relative

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amounts as in Examples 1A and 1B. The beverage had the following composition:

|                                  |                                       | <u>Example</u> |           |
|----------------------------------|---------------------------------------|----------------|-----------|
| <u>INGREDIENTS (% BY WEIGHT)</u> |                                       | <u>2A</u>      | <u>2B</u> |
| 5                                | 80° BEVERAGE ALCOHOL                  | 12.5           | 12.5      |
|                                  | SUCROSE                               | 7.90           | 7.99      |
|                                  | HIGH FRUCTOSE CORN SYRUP              | 8.64           | 8.74      |
|                                  | DEXTROSE (50%)                        | 4.39           | 4.44      |
|                                  | FRUCTOSE (42%)                        | 3.68           | 3.73      |
|                                  | MALTOSE (1.5%)                        | 0.132          | 0.133     |
|                                  | HIGHER SACCHARIDES (5.0%)             | 0.438          | 0.444     |
| 10                               | CITRIC ACID                           | 0.255          | 0.350     |
|                                  | SODIUM CITRATE                        | 0.16           | 0.165     |
|                                  | STABILIZER BLEND                      | 0.089          | 0.090     |
|                                  | FLAVORING                             | 5.49           | 5.524     |
|                                  | DRY COLOR                             | 0.00036        | 0.0144    |
| 15                               | CLOUDING AGENT                        | 0.018          | 0.157     |
|                                  | PRESERVATIVES                         | 0.047          | 0.047     |
|                                  | (POTASSIUM SORBATE & SODIUM BENZOATE) |                |           |
|                                  | BALANCE, DEIONIZED WATER TO 100%      |                |           |

Example 2A yielded a 6.3% wt. alcoholic beverage with a pH of 3.5, having a pineapple flavor. Example 2B yielded a 6.65 % wt. alcoholic beverage with a pH of 3.4, having a frozen margarita flavor.

The flavoring was made as discussed above for the preferred embodiment, i.e., by adding one or more commercial flavors to grain neutral spirits, citric acid and water. The content of flavoring specified above therefore refers to the amount of the resulting mixture of the flavors, grain neutral sprits, citric acid and water.

The product appearance and taste was that of a refreshing frozen cocktail with a very fine crystalline structure.



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EXAMPLES 3A-3C  
(Comparative - Carrageenan As Stabilizing Agent)

In these examples, carrageenan was used in the  
 5 range of 0.1-1.3% by weight instead of the stabilizer  
 blend of the invention. In Example 3A, 0.1% by  
 weight of the carrageenan was used in the alcoholic  
 beverage prepared with the following ingredients,  
 according to the following procedure: 1) carrageenan  
 10 dispersed with sucrose and added to water via high  
 shear mixer; 2) corn syrup added; 3) alcohols, acids,  
 flavorings added.

INGREDIENTS (% BY WEIGHT)

|    |  |       |
|----|--|-------|
|    | OTHER THAN STANDARD ORANGE WINE            | 19.92 |
| 15 | SUCROSE                                    | 7.99  |
|    | HIGH FRUCTOSE CORN SYRUP                   |       |
|    | DEXTROSE (50%)                             | 4.44  |
|    | FRUCTOSE (42%)                             | 3.73  |
|    | MALTOSE (1.5%)                             | 0.133 |
| 20 | HIGHER SACCHARIDES (5.0%)                  | 0.444 |
|    | CITRIC ACID                                | 0.350 |
|    | SODIUM CITRATE                             | 0.165 |
|    | CARRAGEENAN                                | 0.1   |
|    | FLAVORING                                  | 6.41  |
| 25 | CLOUDING AGENT                             | 0.157 |
|    | PRESERVATIVES                              | 0.047 |
|    | (POTASSIUM SORBATE and SODIUM<br>BENZOATE) |       |

BALANCE, DEIONIZED WATER TO 100%

30 In Example 3A, the carrageenan was used as the  
 only stabilizing agent in the amount of 0.1% wt. In  
 Example 3B, the carrageenan was used in the amount of  
 1.3% wt. with 0.10% by weight of carboxymethyl  
 cellulose and in Example 3C the carrageenan was used  
 35 in the amount of 1.3% wt. with 0.25% by weight of  
 CMC. The flavoring in Examples 3A-3C was made as

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discussed above for the preferred embodiment, i.e., by adding one or more commercial flavors to grain neutral spirits, citric acid and water. The content of flavoring specified above therefore refers to the amount of the resulting mixture of the flavors, grain neutral sprits, citric acid and water. In Examples 3B and 3C, the resulting alcoholic beverage was extremely gelled, had undesirable visual appearance, and slimy mouthfeel, as compared to the product of this invention. In Example 3A, the resulting alcoholic beverage had crystalline structure which froze in thin ice crystal sheets, had an undesirable mouthfeel, which was unlike the beverage of the invention. Examples 3A-C yielded a 5.5% wt. alcoholic beverage with a pH of 3.5, having mango flavor.

#### EXAMPLE 4

##### (Comparative - MCC As Stabilizing Agent)

The effect of microcrystalline cellulose ("MCC") as a stabilizing agent was evaluated in this Example. MCC is a naturally occurring pulverized cellulose that has been purified and has been claimed to be a useful stabilizer in frozen foods, particularly for its ability to control ice crystal growth.

The alcoholic beverage was prepared with the following ingredients, according to the following procedure:

Add MCC to water via high shear mixer. Allow to blend 3-4 minutes. Add CMC which has been dispersed with sugar via blender. Add remaining sugars. Mix on stir plate for 15 minutes. Add alcohols and flavors, mix 5 minutes. Add acids.

##### INGREDIENTS (% BY WEIGHT)

|                       |       |
|-----------------------|-------|
| Grain Neutral Spirits | 4.98  |
| OTSW                  | 25.89 |
| HFCS                  | 13.04 |

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|    |                                     |       |
|----|-------------------------------------|-------|
|    | Sugar                               | 9.44  |
|    | Citric Acid                         | 0.26  |
|    | Sodium Citrate                      | 0.19  |
|    | MCC                                 | 1.5   |
| 5  | CMC                                 | 0.2   |
|    | Flavoring                           | 1.689 |
|    | Liquid Color                        | 0.471 |
|    | Cloud                               | 0.12  |
| 10 | BALANCE, DEIONIZED WATER<br>TO 100% |       |

This Example yielded a 10.75% wt. alcoholic beverage with a pH of 3.5, having the flavor of mango. The flavoring included only a commercial  
15 flavor. The content of flavoring specified above therefore refers to the amount of the commercial flavor only.

At the MCC usage levels in the range of 0.5% to about 2.0% by weight suggested in prior art, combined  
20 with CMC at usage levels of 0.1 - 0.4%, the resulting beverage product had a crystalline structure which froze in thin ice crystal sheets, had an undesirable mouthfeel which was unlike the beverage of the invention (small ice crystals, substantial absence of  
25 ice crystal sheets) and also unlike frozen blender drinks.

#### EXAMPLE 5

##### (Comparative - Pectin and CMC As Stabilizing Agent)

Pectin was used in combination with CMC as a  
30 stabilizer blend. Pectin was used in the range of about 0.4% to 0.6% by weight in combination with CMC in the range of 0.01% to 0.04% by weight. The alcoholic beverage was prepared with the following ingredients, according to the following procedure:  
35 1) Using a high shear mixer dissolve pectin and CMC which has been wetted with sugar in water; 2) Let above stir for 10 minutes to insure pectin has

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dissolved; 3) Add sugars, acids and alcohols; 4)  
Flavor and color as desired.

|    |                                     |       |
|----|-------------------------------------|-------|
| 5  | <u>INGREDIENTS (% BY WEIGHT)</u>    |       |
|    | GNS (Grain neutral<br>spirits)      | 4.98  |
|    | OTSW                                | 25.89 |
| 10 | HFCS                                | 13.04 |
|    | Sugar                               | 9.44  |
|    | Citric Acid                         | 0.26  |
|    | Sodium Citrate                      | 0.19  |
| 15 | Pectin                              | 0.5   |
|    | CMC                                 | 0.02  |
|    | Flavoring                           | 1.689 |
| 20 | Liquid Color                        | 0.471 |
|    | Cloud                               | 0.12  |
|    | Balance, Deionized Water<br>To 100% |       |

25

This Example yielded a 10.43% wt. alcoholic  
beverage with a pH of 3.5, having the flavor of  
mango. The flavoring included only a commercial  
30 flavor. The content of flavoring specified above  
therefore refers to the amount of the commercial  
flavor only.

The resulting beverage product, upon freezing,  
had a gelled appearance with ice crystal sheet  
35 formation and undesirable mouthfeel.

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EXAMPLE 6(Comparative - Xanthan Gum As Stabilizing Agent)

5 Xanthan gum was evaluated in the range of about 0.05%  
to about 0.25% by weight alone and in combination  
with the same amount of mannogalactan. The alcoholic  
beverage was prepared with the following ingredients  
and according to the following procedure:

- 1) Mix stabilizer with small amount of sugar
- 10 2) Add above to water via high shear mixer
- 3) Add sugars to above
- 4) Let mixture stir while heating to 165°F
- 5) Cool to 100°F
- 6) Add alcohols and acids - mix for 10 minutes
- 15 7) Add flavorings and colorings - mix for 10 minutes

INGREDIENTS (% BY WEIGHT)

|    |                                     |       |
|----|-------------------------------------|-------|
|    | GNS                                 | 4.98  |
|    | OTSW                                | 25.89 |
|    | HFCS                                | 13.04 |
| 20 | Sugar                               | 9.44  |
|    | Citric Acid                         | 0.26  |
|    | Na Citrate                          | 0.19  |
|    | Xanthan gum                         | 0.15  |
|    | Mannogalactan                       | 0.15  |
| 25 | Flavoring                           | 1.689 |
|    | Liquid Color                        | 0.471 |
|    | Cloud                               | 0.12  |
|    | Balance, Deionized Water<br>to 100% |       |

30

This Example yielded a 10.35% wt. alcoholic  
beverage with a pH of 3.5, having the flavor of  
mango. The flavoring included only a commercial  
flavor. The content of flavoring specified above  
35 therefore refers to the amount of the commercial  
flavor only.

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The resulting beverage product was extremely slimy in mouthfeel and appearance, melted quickly and was therefore undesirable.

5

**EXAMPLE 7**

(Comparative - Sorbitol and Glycerol As Stabilizer Blend)

10

About 1.5% to 2.5% by weight of sorbitol was used in combination with about 0.3% to 0.7% by weight of glycerol as a stabilizer blend.

INGREDIENTS (% BY WEIGHT)

|    |                                     |       |
|----|-------------------------------------|-------|
| 15 | GNS                                 | 4.98  |
|    | OTSW                                | 25.89 |
|    | HFCS                                | 13.04 |
|    | Sugar                               | 9.44  |
|    | Citric Acid                         | .26   |
| 20 | NaCitrate                           | .19   |
|    | Glycerol                            | .5%   |
|    | Sorbitol                            | 2%    |
|    | Flavoring                           | 1.689 |
|    | Liquid Color                        | 0.471 |
| 25 | Cloud                               | 1.2%  |
|    | Balance, Deionized Water<br>to 100% |       |

30

This Example yielded a 10.35% wt. alcoholic beverage with a pH of 3.5, having a flavor of mango. The flavoring included only a commercial flavor. The content of flavoring specified above therefore refers to the amount of the commercial flavor only.

35

The alcoholic beverage was prepared with the following ingredients, according to the following procedure:

- 1) Mix glycerol and sorbitol with sugar in water via high shear mixer.

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- 2) Stir for 15 minutes.
- 3) Add alcohol and acids, stir for 10 minutes.
- 4) Add flavorings and colors, stir for 10 minutes.

The resulting beverage product, after freezing, melted extremely fast and required lower freezer temperatures not consistently and reliably achievable by home freezers.

#### EXAMPLE 8

10        (Comparative - CMC Alone Or With Other  
          Ingredients As Stabilizing Agent)

15        This Example summarizes an experimental design for formulation change of various stabilizers (or stabilizing ingredients). The parameters of the design are summarized below including the data of Table A.

20        In particular, the use of CMC was evaluated both alone and in combination with a variety of other stabilizing ingredients, such as MCC, pectin, guar gum, locust bean gum. The beverages of this Example had pH of about 3.2 - about 3.3, alcohol content of about 6.1 - about 6.4, % wt., and margarita flavor.

25        Eight formulations involving varying levels of four gums and stabilizers were evaluated along with a beverage formulation comprising locust bean gum, guar gum and pectin, used as a control, and a modified mix of stabilizers (no CMC) [Table A].

30        The basic formulation for the beverages is summarized below, and details of the stabilizer blends of the eight formulas used in this experimental design are listed in Table A.

#### INGREDIENTS (% BY WEIGHT)

|   |        |
|---|--------|
| 35        Other Than Standard Orange Wine |        |
| (21% ethyl alcohol)                       | 19.63% |
| Sucrose                                   | 7.71%  |
| High Fructose Corn Syrup                  |        |

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|    |   |        |
|----|---|--------|
|    | Dextrose (50%)  | 4.25   |
|    | Fructose (42%)  | 3.57   |
|    | Maltose (1.5%)  | 0.1275 |
|    | Higher Saccharides  | 0.425  |
| 5  | Citric Acid   | 0.33   |
|    | Sodium Citrate  | 0.159  |
|    | Stabilizer  |        |
|    | Flavoring   | 6.42   |
|    | Clouding Agent  | 0.14   |
| 10 | Liquid Color  | 0.089  |
|    | Preservatives   | 0.046  |
|    | Beverage total % alcohol by weight: about 6.1 - about 6.4%.   |        |
|    | Beverage pH: about 3.2 - about 3.3.   |        |
| 15 | The flavoring was made as discussed above for the preferred embodiment, i.e., by adding one or more commercial flavors to grain neutral spirits, citric acid and water. The content of flavoring specified above therefore refers to the amount of the resulting mixture of the flavors, grain neutral sprits, citric acid and water. |        |
|    |   |        |
|    |   |        |
| 20 |   |        |

Table A -- Stabilizer Information

| FORMULA | GUAR GUM | LOCUST BEAN GUM | CMC   | LMP  |
|---------|----------|-----------------|-------|------|
| 1       | 0.020    | 0.025           | 0.0   | 0.15 |
| 2       | 0.015    | 0.036           | 0.0   | 0.15 |
| 3       | 0.015    | 0.025           | 0.018 | 0.15 |
| 4       | 0.020    | 0.036           | 0.018 | 0.15 |
| 5       | 0.015    | 0.025           | 0.0   | 0.60 |
| 6       | 0.020    | 0.036           | 0.0   | 0.60 |
| 7       | 0.020    | 0.025           | 0.018 | 0.60 |
| 8       | 0.015    | 0.036           | 0.018 | 0.60 |



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Eight panelists were trained in descriptive analysis techniques to evaluate frozen pouched products. Panelists were broken up into two teams of four where the team members each evaluated a sample from the same pouch. Three test samples and the control were evaluated during each session. Panelists independently rated each sample. The results are summarized in Table 2. The results indicate that, while CMC had been used largely in prior art, it was found to not be effective in the beverage of this invention. Table 2 results also indicate that among the four evaluated gums and stabilizers, the research suggests that CMC has the least effect from formulation changes on the sensory profile of the product. LMP had the greatest effects, even at its lowest use levels. Further experimentation indicated that LMP's optimum use level had not been achieved. Such optimum level was found to be much lower than the lowest level used in Example 8. As disclosed herein, the optimum LMP level is about 0.003 to about 0.1% by weight. The CMC's least effect on the sensory profile of the product indicated in Table 2 is believed to be due largely because CMC is functional in higher pH systems, especially those in the range of pH 4.0-10.0.

Furthermore, as shown in Table 1, surprisingly, in an experiment conducted with various stabilizing ingredients (i.e., locust bean gum, guar gum, LMP), CMC was found to have the least effect on formulation changes in this invention, i.e., removing CMC from formula had no effect on visual crystal size, rate of meltdown, visual sliminess, mouthfeel ice crystal size, mouthfeel ice crystal sheet size, mouthfeel sliminess, crunch or gumminess.

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Without wishing to be bound by any operability theory, it is believed that the preferred embodiment of the beverage of this invention has a considerably lower pH, than the preferred range for CMC, and therefore CMC is substantially non-functional in the pH system of this embodiment of our invention. It is believed that CMC is unable to control crystalline growth and deliver a superior texture once frozen at these pH levels.

10

EXAMPLES 9A-9B  
(Beverage of the Invention)

|                                  |   | <u>Example</u> |           |
|----------------------------------|---|----------------|-----------|
| <u>INGREDIENTS (% BY WEIGHT)</u> |   | <u>9A</u>      | <u>9B</u> |
|                                  | OTHER THAN STANDARD ORANGE WINE         | 19.71          | 19.92     |
|                                  | SUCROSE                                 | 7.90           | 7.99      |
| 15                               | HIGH FRUCTOSE CORN SYRUP                |                |           |
|                                  | DEXTROSE (50%)                          | 4.39           | 4.44      |
|                                  | FRUCTOSE (42%)                          | 3.68           | 3.73      |
|                                  | MALTOSE (1.5%)                          | 0.132          | 0.133     |
|                                  | HIGHER SACCHARIDES (5.0%)               | 0.438          | 0.444     |
|                                  | CITRIC ACID                             | 0.255          | 0.350     |
| 20                               | SODIUM CITRATE                          | 0.16           | 0.165     |
|                                  | STABILIZER BLEND                        | 0.089          | 0.090     |
|                                  | FLAVORING                               | 5.49           | 5.524     |
|                                  | DRY COLORING                            | 0.00036        | 0.0144    |
|                                  | CLOUDING AGENT                          | 0.018          | 0.157     |
|                                  | PRESERVATIVES                           | 0.047          | 0.047     |
| 25                               | (POTASSIUM SORBATE and SODIUM BENZOATE) |                |           |
|                                  | BALANCE, DEIONIZED WATER                |                |           |

Example 9A yielded a 5.5% wt. alcoholic beverage with a pH of 3.5, having the flavor of pineapple, and Example 9B yielded a 5.82% wt. alcoholic beverage with pH of 3.4, having the flavor of margarita.

In Example 9A, the stabilizer blend was comprised of 0.035% by weight of locust bean gum, 0.018% by weight of guar gum and 0.018% by weight of CMC and a filler as balance. In Example 9B, the

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stabilizer blend was comprised of 0.018 guar gum and 0.0495 locust bean gum and a filler as a balance. The flavoring used was that suitable for a frozen margarita type cocktail, and additional ingredients included: a clouding agent and preservatives, such as sodium benzoate and potassium sorbate. The flavoring was made in Examples 9A and 9B as discussed above for the preferred embodiment, i.e., by adding one or more commercial flavors to grain neutral spirits, citric acid and water. The content of flavoring specified above therefore refers to the amount of the resulting mixture of the flavors, grain neutral sprits, citric acid and water. Product characteristics and evaluation of Examples 9A and 9B can be found under Example 10 and Table 1.

#### EXAMPLE 10

(Taste Panel Testing)

Properties of a beverage product produced according to Example 1 of Ashmont et al., U.S. Patent No. 4,790,999 (using sodium CMC as a stabilizer), were compared to a control product, i.e., made according to Example 9A. The product of Example 9A contained a blend of guar gum, locust bean gum and CMC as a stabilizer. The beverage of Example 9A was compared to the beverage of Example 9B. The beverage of Example 9B contained no CMC, but used guar gum and locust bean gum as a stabilizer. The results are summarized in Table 1.

The effects of different stabilizers on the sensory profile of the frozen beverage product made according to Examples 9A and 9B were evaluated by eight panelists trained in descriptive analysis techniques to evaluate frozen pouched products.

Substantial sensory differences were found between Ashmont's Example 1 and Example 9A. The product of Ashmont's Example 1 had larger visual ice

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crystal sheet size, quicker rate of melt, larger ice crystal mouthfeel size, larger ice crystal sheet mouthfeel size and more crunch.

5 A modified version of the Example 9A beverage, comprising guar and locust bean gums as the stabilizer blend, i.e., Example 9B, was compared to the beverage of Example 9A. No significant differences were detected by panelists. This is believed to be because CMC is substantially  
10 non-functional in low pH systems. The results are summarized in Table 1.

TABLE 1. SUMMARY OF DIFFERENCES BETWEEN BEVERAGES PRODUCED ACCORDING TO EXAMPLES 9A AND 9B AND ASIMONT'S EXAMPLE 1.

| COMPARISON                                      | ATTRIBUTE VS. CONTROL |                   |             |                  |                          |                        |                       |           |           |
|---|-----------------------|-------------------|-------------|------------------|--------------------------|------------------------|-----------------------|-----------|-----------|
|   | VISUAL CRYSTAL SIZE   | VISUAL SHEET SIZE | MELTDOWN    | VISUAL SLIMINESS | MOUTHI FEEL CRYSTAL SIZE | MOUTHI FEEL SHEET SIZE | MOUTHI FEEL SLIMINESS | CRUNCH    | GUMMINESS |
| Asimont (CMC) vs. Ex. 9A (Guar gum, LBG, CMC)   | (N5)                  | Larger (**)       | Faster (**) | (N5)             | Larger (**)              | Larger (**)            | (N5)                  | More (**) | (N5)      |
| Ex. 9D (no CMC) vs. Ex. 9A (Guar gum, LBG, CMC) | (N5)                  | Smaller (*)       | (N5)        | (N5)             | (N5)                     | (N5)                   | (N5)                  | (N5)      | (N5)      |

\* Directional (&lt;20% type I error)

\*\* Significant (&lt;10% type I error)

NS Not Significant (&gt;20% type I error)

LBG Locust Bean Gum

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As shown in Tabl 1, there are major sensory differences between the Example 9A beverage and the Ashmont's formula (Table 1). The Example 9A formulation had smaller ice crystal sheets and melted at a significantly slower rate. A formulation without CMC, i.e., Example 9B (Table 1), appears promising as an improvement over beverages containing guar gum, locust bean gum and cellulose gum.

TABLE 2. SUMMARY OF THE EFFECTS OF STABILIZER ON THE SENSORY PROFILE OF FROZEN POUCHED PRODUCT.

| EFFECT          | ATTRIBUTE           |                   |          |                 |                        |                      |                    |        |               |
|-----------------|---------------------|-------------------|----------|-----------------|------------------------|----------------------|--------------------|--------|---------------|
|                 | VISUAL CRYSTAL SIZE | VISUAL SHEET SIZE | MELTDOWN | VISUAL SLIMNESS | MOUTHFEEL CRYSTAL SIZE | MOUTHFEEL SHEET SIZE | MOUTHFEEL SLIMNESS | CRUNCH | GUMMINESS     |
| Quar Gum        | Decrease (*)        | (NS)              | (NS)     | Increase (**)   | (NS)                   | (NS)                 | Increase (**)      | (NS)   | (NS)          |
| Locust Bean Gum | (NS)                | (NS)              | (NS)     | Increase (**)   | (NS)                   | (NS)                 | (NS)               | (NS)   | (NS)          |
| CMC             | (NS)                | (NS)              | (NS)     | Increase (**)   | (NS)                   | (NS)                 | (NS)               | (NS)   | (NS)          |
| LMP             | (NS)                | (NS)              | (NS)     | Increase (**)   | Increase (**)          | Increase (**)        | Increase (**)      | (NS)   | Increase (**) |

\* Directional (&lt; 20% type I error)

\*\* Significant (&lt;10% type I error)

NS Not Significant (&gt;20% type I error)

LMP Low Methoxy Pectin

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EXAMPLE 11(Experimental Design)

In this experimental design, the beverage of the invention was further optimized by evaluating a set of formulas with varying levels of LMP, GG and LBG. Also formulated were a product of the invention (Example 9A) (CONTROL) and the Example 1A of Ashmont et al. both including CMC. These products were evaluated by a trained descriptive panel with all products seen in random order. Formulations were evaluated for nine descriptive attributes as in the studies summarized in Tables 1 and 2.

One of the objectives was to determine the advantages of using LMP in a formula where CMC is absent. Another objective was to optimize the invention product while reducing LBG to create a cost savings opportunity.

Product Optimization

Formulations are presented in Table 3. Three descriptive attributes were significant among the set of 18 products formulated by varying LMP, GG and LBG (visual sliminess, crystal sheet size texture and crunchiness). Conceptually, an ideal product is believed to minimize visual sliminess and sheet size, while maximizing crunchiness. Locust bean gum had the greatest effect on visual sliminess. Minimal sliminess could be maintained with formulations at the lowest level of LBG and different combinations of LMP and GG. Sheet size texture was affected by LMP interactions with both LBG and GG. Sheeting of ice crystals was minimized by formulations of high LBG with low LMP and low amounts of GG, and by formulations of low LBG with high LMP and moderate amounts of GG.



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Crunchiness was maximized through an interaction between LBG and GG. The level of LMP had no significant effect on crunchiness although crunchiness appeared to be enhanced at the intermediate level of LMP and highest level of GG.

#### Results

These results suggest the invention product can be sufficiently optimized using LMP to reduce LBG and provide a cost savings opportunity. At the lowest levels of LBG increasing LMP and decreasing GG minimized sliminess and ice sheeting. Also at the lowest levels of LBG crunchiness was maximized by increasing both GG and LMP. Therefore, a cost effective optimized formula includes a reduced level of LBG, a relatively high level of LMP and an intermediate level of GG.

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**TABLE 3. EXPERIMENTAL PLAN**

| EXPERIMENTAL DESIGN<br>ORDER FOR SENSORY EVALUATIONS |             |                   |                          |
|--|-------------|-------------------|--------------------------|
| TRIAL  | GUAR<br>GUM | LOCUS<br>BEAN GUM | LOW<br>METHOXY<br>PECTIN |
| 1  | 0.015000    | 0.047500          | 0.012500                 |
| 6  | 0.020000    | 0.047500          | 0.025000                 |
| 15   | 0.020000    | 0.047500          | 0.012500                 |
| 15   | 0.020000    | 0.047500          | 0.012500                 |
| 9  | 0.022887    | 0.034510          | 0.019717                 |
| 13   | 0.022887    | 0.034510          | 0.005283                 |
| 4  | 0.020000    | 0.070000          | 0.012500                 |
| 15   | 0.020000    | 0.047500          | 0.012500                 |
| 14   | 0.017113    | 0.034510          | 0.005283                 |
| 7  | 0.022887    | 0.060490          | 0.019717                 |
| 8  | 0.017113    | 0.060490          | 0.019717                 |
| 2  | 0.025000    | 0.047500          | 0.012500                 |
| 15   | 0.020000    | 0.047500          | 0.012500                 |
| 12   | 0.017113    | 0.060490          | 0.005283                 |
| 5  | 0.020000    | 0.047500          | 0.000000                 |
| 10   | 0.017113    | 0.034510          | 0.019717                 |
| 3  | 0.020000    | 0.025000          | 0.012500                 |
| 11   | 0.022887    | 0.060490          | 0.005283                 |

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**EXAMPLE 12**  
**(Experimental Design)**

5 A study was conducted to determine how locust  
bean gum (LBG), guar gum (GG) and low methoxy pectin  
(LMP) function within the product of the invention.  
10 Descriptive analysis was used to characterize the  
changes in product attributes as these three gums  
were varied. Three descriptive attributes were  
"mouthfeel-sheet size" and "crunchiness". An ideal  
15 product for consumer acceptance is believed to  
minimize the attributes "sliminess" and "mouthfeel-  
sheet size" and have an intermediate level of  
"crunch." As the relative importance of each  
15 attribute is not known to drive consumer acceptance,  
different responses were evaluated which represented  
different weights on these three descriptive  
attributes.

20 **CONCLUSIONS**

Four different "ideal product" formulations were  
prepared, assuming "crunchiness" to contribute  
different weights to maximize consumer acceptance.  
The respective optimum levels for "ideal" are  
25 presented in Table B. As "crunch" becomes less of a  
factor driving consumer acceptance (a softer product  
is preferred), a larger amount of low methoxy-pectin  
is needed to optimize. As "crunchiness" is more of a  
factor in driving consumer acceptance, then a formula  
30 with high LBG and GG, and low LMP will achieve the  
optimum.

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Table B. Ideal formulations under different assumptions about the relative importance of "crunchiness" to consumer acceptance.

| IDEAL PRODUCT ATTRIBUTE | Low Methoxy<br>Pectin (w/v) | Guar Gum<br>(w/v) | Locust Bean<br>Gum (w/v) |
|-------------------------|-----------------------------|-------------------|--------------------------|
| 100% CRUNCHINESS        | 0.0053                      | 0.0229            | 0.0345                   |
| 33% CRUNCHINESS         | 0.0053                      | 0.0229            | 0.0345                   |
| 11% CRUNCHINESS         | 0.0053                      | 0.0229            | 0.0345                   |
| 0% CRUNCHINESS          | 0.0197                      | 0.0171            | 0.0345                   |

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**EXAMPLE 13**  
**(Container of the Invention)**

A flexible pouch container having the Structure C was prepared. The pouch contained the following layers, with the layer 1 being the outer film, and layer 3 the film in contact with the beverage.

**Structure C**

Layer 1. About 0.0005 inch to about 0.0007 inch biaxially oriented Nylon;

Layer 2. About 0.0006 inch to about 0.0008 inch aluminum foil;

Layer 3. About 0.0020 inch to about 0.0040 inch copolymer of ethylene and propylene and/or polyethylene.

**EXAMPLES 14A - 14B**  
**(Container of the Invention)**

Two other flexible pouches of the invention were prepared to produce the pouches of Structures A and B, summarized below.

**Structure A - Example 14A**

Layer 1. About 0.00040 inch to about 0.00060 inch polyester;

Layer 2. About 0.0004 inch to about 0.0006 inch aluminum foil;

Layer 3. About 0.00040 inch to about 0.00060 inch polyester;

Layer 4. About 0.0020 inch to about 0.0040 inch linear low density polyethylene or linear low density polyethylene which includes octene and ethylene.

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Structure B - Example 14B

Layer 1. About 0.0060 inch to about 0.0080 inch polyvinylidene chloride coated biaxially oriented Nylon;

5 Layer 2. About 0.0009 inch to about 0.0011 inch Nylon/ethylenevinyl alcohol coextrusion;

Layer 3. About 0.0020 inch to about 0.0030 inch linear low density polyethylene (homopolymer).

EXAMPLES 15-19

10 (Testing of Containers)

The strength of the pouch of Example 13 was compared to that of Examples 14A and 14B. All pouches had substantially the same size and contained approximately 200 ml of the alcoholic beverage of the  
15 invention.

The pouches were drop tested in a shipping case and shelf life tests were performed as indicated in the following data table.

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| Example No. | Container of Example | Structure   | NSTA Drop Test* | Shelf Life Week |
|-------------|----------------------|---|-----------------|-----------------|
| 15          | 13                   | Polypropylene, no pads                            | 4               | 52              |
| 16          | 13                   | Polypropylene, 2 pads**                           | 8               | 52              |
| 17          | 13                   | Polypropylene, 2 pads and plastic bubble sheet*** | 10              | 52              |
| 18          | 14A                  | Polyethylene, no pads                             | 10              | 15              |
| 19          | 14B                  | Polyethylene, no pads                             | 23              | 10              |

\* Data reported -- number of successful drops before 1st pouch burst. Failure is due to leaking pouches.

\*\* The structure of Example 13 with two open faced A-C flute doublewall corrugated pads positioned beneath pouches in the shipcase. The total pad thickness was 0.75 inch.

\*\*\* The structure of Example 13 with 0.5 inch thick plastic bubble sheet positioned between two open faced A-C flute doublewall corrugated pads positioned beneath pouches in the shipcase. The total pad thickness was 1.25 inch.

The droptest procedure used was as follows:

National Safe Transit Association Preshipment Testing Procedure 1A Droptest 1 through 20.99 pounds.

This testing is used to predetermine the probability of the successful delivery of packaged products to their destination. Five shipcases of product are subjected to a total 10 drops from 30" off floor (til over concrete). The procedure for the drop test is listed in a specific methodical order so that impacts occur on each side of box, one corner,

SUBSTITUTE SHEET (RULE 26)

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and 3 edges. The 5 shipcases are opened and the contents are inspected for damage.

Results

1. Changing the inside plastic film lamination  
5 layer next to the product from polypropylene  
(Examples 15-17) to polyethylene (Examples 18-



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- 19) yielded improved drop characteristics. Drop failures were believed to be due to a liquid hydraulic force upon the sides of the pouch. Polypropylene (Example 13) with no pads was unacceptable due to the high likelihood of damage in distribution and transportation. A cost savings can be realized with the elimination of cushion pads used in Examples 16 and 17. The polyethylene pouch (Examples 18 and 19) required no pads to achieve the minimum NSTA requirement of 10 drops with no broken pouches and leaking product. The performance of pouch hydraulic failure resistance of Examples 18 and 19 was equal to or greater than that of the pouch of Example 17.
2. Shelf life was not compromised by using polyethylene lamination next to the product in place of polypropylene at freezer, room and oven temperatures. The pouch of Example 14A achieved 15 weeks of successful shelf life with no loss of flavor preference determined by sensory taste panelists. The pouch of Example 14B achieved 10 weeks of successful shelf life with no loss of flavor preference determined by sensory taste panelists. Therefore, polyethylene as a plastic laminate next to the product does not affect flavor as indicated in prior art.
3. The non-aluminum foil structure of Example 14B has superior flex crack resistance when subjected to vibration motion testing. No visual signs of cracking, bruising, or creasing were evident. The foil structures of Examples 13 and 14A may crease, wrinkle and possibly leak product from severe flex cracks under severe vibration testing as would occur in truck or

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rail transportation. Our testing indicated that shelf life performance was not dependent on foil as the only form of barrier material for the alcoholic beverage product of the invention.

5 Also, we would expect that the plastic film could be metalized as is commonly applied in the packaging industry and this could be utilized in place of aluminum foil lamination.

The embodiments described above provide a number  
10 of significant advantages. The ready-to-freeze alcoholic beverage of the invention has a higher pH level which has been previously thought in prior art to be more compatible with CMC but not with pectin. It has excellent shelf stability of at least one  
15 year, which enables wholesale and retail establishments to maintain it in stock for prolonged time periods, thereby minimizing the necessity to rotate the stock. The beverage provides a very convenient method for a consumer to produce a frozen  
20 cocktail in a relatively short time if containers containing the beverage are stored in consumer's freezer. Yet, the beverage, when frozen, has excellent organoleptic properties very similar to those of such beverages made to order from individual  
25 ingredients. The frozen beverage, once removed from the freezer, also maintains its semi-frozen, slushy consistency for a prolonged period of time.

It will be apparent to those skilled in the art that specific embodiments discussed above can be  
30 successfully repeated with ingredients equivalent to those generically or specifically set forth above and under variable process conditions.

It is therefore intended that the foregoing detailed description be regarded illustrative rather  
35 than limiting, and that it be understood that it is

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the following claims, including all equivalents,  
which are intended to define the scope of this  
invention.

**CLAIMS:**

- 1           1.   A ready-to-freeze alcoholic beverage having  
2   pH of about 3.0 to about 5.0, proof of about 6 to  
3   about 28 and comprising:
  - 4               a.   a beverage alcohol;
  - 5               b.   a stabilizer blend comprised of: locust  
6   bean gum and guar gum;
  - 7               c.   a flavoring;
  - 8               d.   at least one sugar; and
  - 9               e.   deionized water.
- 1           2.   An alcoholic beverage of claim 1, wherein  
2   the stabilizer additionally comprises pectin.
- 1           3.   An alcoholic beverage of claim 1 having  
2   proof of about 8 to about 14.
- 1           4.   An alcoholic beverage of claim 2 having  
2   proof of about 8 to 14.
- 1           5.   An alcoholic beverage of claim 1 having  
2   proof of about 11 to about 13.
- 1           6.   An alcoholic beverage of claim 2 having  
2   proof of about 11 to about 13.
- 1           7.   An alcoholic beverage of claim 1 comprising  
2   about 0.04 to about 0.13% by weight of the stabilizer  
3   blend.
- 1           8.   An alcoholic beverage of claim 1, wherein  
2   the sugar comprises sucrose, fructose and dextrose.
- 1           9.   An alcoholic beverage of claim 1, wherein  
2   the sugar comprises sucrose and high fructose corn  
3   syrup.
- 1           10.   An alcoholic beverage of claim 9 wherein  
2   the high fructose corn syrup comprises dextrose,  
3   fructose, maltose and higher saccharides.
- 1           11.   An alcoholic beverage of claim 1, wherein  
2   the beverage alcohol is a monohydric beverage  
3   alcohol.

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1           12. An alcoholic beverage of claim 1, wherein  
2 the beverage alcohol is provided by an alcoholic  
3 beverage selected from the group consisting of grain  
4 neutral spirits, vodka, whisky, rum, tequila, wine,  
5 malt beverage and Other Than Standard wine.

1           13. An alcoholic beverage of claim 12, wherein  
2 the Other Than Standard wine is an orange or other  
3 fruit source Other Than Standard wine.

1           14. An alcoholic beverage of claim 1 comprising  
2 sugar content measured in Brix of about 12 to about  
3 19°.

1           15. An alcoholic beverage of claim 1, which  
2 comprises, as the stabilizer blend, about 0.025 to  
3 about 0.090% by weight of locust bean gum and about  
4 0.005 to about 0.5% by weight of guar gum.

1           16. An alcoholic beverage of claim 15, which  
2 comprises, as the stabilizer blend, about 0.030 to  
3 about 0.06% by weight of locust bean gum and about  
4 0.010 to about 0.30% by weight of guar gum.

1           17. An alcoholic beverage of claim 1, which  
2 consists essentially of about 0.025 to about 0.090%  
3 by weight of locust bean gum and about 0.005 to about  
4 0.5% by weight of guar gum, as the stabilizer blend.

1           18. An alcoholic beverage of claim 2, which  
2 comprises, as the stabilizer blend, about 0.025 to  
3 about 0.090% by weight of locust bean gum, about  
4 0.005 to about 0.5% by weight of guar gum and about  
5 0.003 to about 0.1% by weight of pectin.

1           19. An alcoholic beverage of claim 18, which  
2 comprises as the stabilizer blend, about 0.030 to  
3 about 0.06% by weight of locust bean gum, about 0.010  
4 to about 0.30% by weight of guar gum and about 0.003  
5 to about 0.075% by weight of pectin.

1           20. An alcoholic beverage of claim 2, which  
2 consists essentially of about 0.025 to about 0.090%

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3 by weight of locust bean gum, about 0.005 to about  
4 0.5% by weight of guar gum, and about 0.003 to about  
5 0.1% by weight of pectin, as the stabilizer blend.

1 21. An alcoholic beverage of claim 1, which  
2 additionally comprises more than one flavoring agent.

1 22. An alcoholic beverage of claim 1, which  
2 additionally comprises at least one acidulant.

1 23. A method of preparing a ready-to-freeze  
2 alcoholic beverage comprising:

3 a. blending a stabilizer blend, comprised  
4 of a locust bean gum and guar gum, with about 5-20%  
5 of total dry sugar used in the beverage;

6 b. adding the resulting mixture to  
7 approximately 25% portion of a water phase,  
8 comprising deionized water;

9 c. adding the remainder of the dry sugar  
10 to produce a sugar/stabilizer slurry;

11 d. heating the sugar/stabilizer slurry to  
12 a temperature of about 149°- about 190°F;

13 e. cooling the slurry to a temperature of  
14 about 100°F; and

15 f. adding the resulting cooled slurry to a  
16 balance of the alcoholic beverage formulation  
17 comprising: deionized water, a flavoring and a  
18 beverage alcohol.

1 24. A method of claim 23, wherein the  
2 stabilizer blend comprises pectin.

1 25. A method of claim 23, wherein in the step  
2 e. the balance of the alcoholic beverage comprises a  
3 preservative, a clouding agent, an acidulant and a  
4 colorant.

1 26. A container comprising the following multi-  
2 layered structure:

3 (a) polyester film;

4 (b) aluminum foil;

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5 (c) polyester film;

6 (d) linear low density polyethylene film  
7 or linear low density polyethylene which includes  
8 octene and ethylene.

1 27. A container of claim 26 additionally  
2 comprising a layer of an adhesive between each of the  
3 layers.

1 28. A container of claim 26, wherein the layer  
2 (a) has the thickness of about 0.00040 - about  
3 0.00060 inches; the layer (b) has the thickness of  
4 about 0.0004 - about 0.0006 inches; the layer (c) has  
5 the thickness of about 0.00040 - about 0.00060  
6 inches; and the layer (d) has the thickness of about  
7 0.0020 - about 0.0040 inches.

1 29. A container of claim 26, wherein said  
2 polyester film is reverse gravure printed.

1 30. A container comprising the following multi-  
2 layered structure:

3 (a) a Nylon film;

4 (b) a Nylon/ethylene/vinyl alcohol co-  
5 extruded layer film;

6 (c) a linear low density polyethylene  
7 film.

1 31. A container of claim 30 additionally  
2 comprising a layer of an adhesive between each of the  
3 layers.

1 32. A container of claim 30, wherein the layer  
2 (a) has the thickness of about 0.0060 - about 0.0080  
3 inches; the layer (b) has the thickness of about  
4 0.0009 - about 0.0011 inches; and the layer (c) has  
5 the thickness of about 0.0020 - about 0.0030 inches.

1 33. The container of claim 30, wherein said  
2 Nylon film is reverse gravure printed.

1 34. A container comprising the following multi-  
2 layered structure:

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3           (a) a Nylon film;  
4           (b) aluminum foil;  
5           (c) a copolymer of ethylene and propylene  
6 film.

1       35. A container of claim 34 additionally  
2 comprising a layer of adhesive between each of the  
3 layers.

1       36. A container of claim 34, wherein the layer  
2 (a) has the thickness of about 0.0005 - about 0.0007  
3 inches; the layer (b) has the thickness of about  
4 0.0006 - about 0.0008 inches; and the layer (c) has  
5 the thickness of about 0.0020 - about 0.0040 inches.

1       37. The container of claim 34, wherein said  
2 Nylon film is reverse gravure printed.



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US95/13242

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) :A22C 13/00; B32B 27/04, 27/08; C08G 63/02; C12G 3/04

US CL :Please See Extra Sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/115, 592; 427/393.5; 428/34.8, 35.4, 36.6, 36.7, 353, 423.5, 518; 528/272, 274

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                | Relevant to claim No. |
|-----------|---|-----------------------|
| Y         | US, A, 4,790,999 (ASHMONT ET AL) 13 December 1988, col. 2, lines 54-68, col. 3, lines 21-53, col. 4, lines 19-38. | 1-25                  |
| Y         | US, A, 3,826,829 (MARULICH) 30 July 1974, col. 2, lines 63-64, Example 1.   | 1-25                  |
| Y         | US, A, 5,246,753 (KOYAMA ET AL) 21 September 1993, col. 2, lines 58-68, col. 4, lines 7-26, col. 7, lines 38-40.  | 26-37                 |
| Y         | US, A, 4,927,689 (MARKIEWICZ) 22 May 1990, col. 5, lines 9-45, col. 6, lines 58-62, col. 7, lines 33-34.          | 26-37                 |
| A         | US, A, 5,085,895 (ASANUMA ET AL) 04 February 1992.  |                       |

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

|   |    |  |
|---|----|--|
| * Special categories of cited documents:  | T  | later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principles or theory underlying the invention   |
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| *E* earlier document published on or after the international filing date  | Y* | document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art |
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| *O* document referring to an oral disclosure, use, exhibition or other means  |    |  |
| *P* document published prior to the international filing date but later than the priority date claimed  |    |  |

Date of the actual completion of the international search

04 JANUARY 1996

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# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US95/13242

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|--|-----------------------|
| A         | US, A, 4,820,795 (HIRATA ET AL) 11 April 1989.                                     |                       |

**INTERNATIONAL SEARCH REPORT**

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**A. CLASSIFICATION OF SUBJECT MATTER:**

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426/115, 592, 427/393.5; 428/34.8, 35.4, 36.6, 36.7, 353, 423.5, 518; 528/272, 274